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A Process to Regenerate the Low-Temperature Condensation Surfaces of a Cryopump and a Cryopump Device to Implement the Process.

To shorten the regeneration time of two-stage autonomous cryopumps, the thermal contact between the condensation surfaces and the low-temperature stage of the cryogenerator is eliminated during the regeneration, where the latter remains operational. Furthermore, defrosting is suitably effected by a heating element that is hermetically separated from the pump chamber.

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## CLAIMS

1. A process to regenerate the low-temperature condensation surfaces of an autonomous cryopump that is cooled by a cryogenerator, characterized as follows: Before defrosting the gases that have condensed on the condensation surfaces, the thermal contact between the cryogenerator and the condensation surfaces is eliminated.

2. A process according to Claim 1 characterized as follows: After the thermal contact between the cryogenerator and the cryosurfaces has been eliminated, the cryosurfaces are heated with a heating element while the cryogenerator remains in operation.

3. A cryopump device to perform the process according to Claim 1 with an autonomous cryopump, a cryogenerator, and with low-temperature condensation surfaces for the bound gases, where these condensation surfaces are cooled by the cryogenerator during pumping, characterized as follows: The thermal contact between the cryogenerator and the condensation surfaces is designed so that it can be established or eliminated at will.

4. A cryopump device according to Claim 3 characterized by the cryopump having a two-stage design, in such a fashion that both the elimination and reestablishment of thermal contact between the low-temperature stage and the associated cryosurfaces, as well as the establishment and re-elimination of the thermal contact between these cryosurfaces and the high-temperature stage is possible.

5. A cryopump device according to Claim 3 characterized by the cryopump containing an electrical heating device for the cryosurfaces.

6. A cryopump device according to Claim 5 characterized by the chamber, in which the heating body and the associated cryogenerator stage are located, being evacuable.

The invention refers to a process for regenerating the low-temperature condensation surfaces of a cryopump according to the definition of the species of Claim 1, and to a cryopump device to implement the process. Cryopumps are used to generate fine and high vacuum in vacuum systems, in which vacuum processes are carried out on an industrial scale. For some years, cryopumps have been increasingly used for this purpose, since they not only have a very high specific suction power, but can also generate a "clean" vacuum, free from hydrocarbons with low-end pressures. In contrast to conveyance-type vacuum pumps, the suctioned-off gases are stored in the cryopump. Consequently, regeneration is necessary from time to time. The invention is especially concerned with this problem.

Cryopumps are used, for example, in systems to produce thin layers by means of cathode atomization. These systems work with a relatively large argon throughput in the pressure range from $10^{-3}$ to $10^{-2}$ mbar. In order to achieve reproducible results and a good layer quality with program-controlled systems, the partial pressure of the remaining residual gases, especially the partial pressure of hydrogen, must be kept as low as possible in the coating chamber.

However, an adequate suction capacity for hydrogen is also important with other vacuum processes, e.g. with high-vacuum sputtering, in the vaporization of metals, whether from a vaporization boat and vaporization material or whether through wall reactions in the vacuum chamber. In all these processes, considerable quantities of hydrogen are sometimes liberated. But the storage capacity of customary cryopumps is low precisely for hydrogen - also for helium.

Cryogenerators are currently generally used to operate so-called autonomous cryopumps, meaning cryopumps which operate without an external supply of coolant. Such pumps are based either on the Stirling cycle or on the Gifford-McMahon cycle. To achieve the low temperatures that are necessary to condense the permanent gases, there are two cryogenerator stages connected sequentially. The cryocondensation surfaces that are associated with the first stage will be referred to below as the high-temperature stage (HT stage). The more easily condensible gases, such as water vapor, $CO_2$, and higher hydrocarbons are condensed there. Its temperature generally lies in the range of 70-120 K. It simultaneously cools the radiation shielding for the second stage, which will be referred below as the low-temperature stage (LT stage). The condensation surfaces connected therewith freeze out gases such as Ar, $O_2$, and $N_2$ or, by means of cryosorption, they bind gases such as $H_2$, He, and Ne to an absorbing agent, for example activated charcoal. The temperature of the LT stage generally lies at 15-20 K.

The temperature which establishes itself at the cryosurfaces of the two stages is determined on the one hand by the cryogenic power available respectively at the two stages and, on the other hand, by the enthalpy of the suctioned gases and by the heat flow which is brought in from the environment by radiation and heat conduction.

The equilibrium pressure of the condensed or absorbed gases, e.g. hydrogen, is a function of the temperature which establishes itself at the LT stage. Although $H_2$ has an equilibrium pressure of about 1 bar at a temperature of 20 K, it is possible to reduce the hydrogen partial pressure to below $10^{-6}$ mbar by means of cryosorption on activated charcoal which is glued onto the LT cryosurface. The amount of hydrogen that can be pumped thereby is limited,

however. It depends on the quantity of absorbing agent, its temperature, and on the quantity of other gases which are absorbed at the same time or which already have been absorbed previously. After some time, the absorbing agent saturates, and the equilibrium pressure of hydrogen begins to rise. Then it is necessary to regenerate the absorbing agent by out-heating. Up to now, this has only been possible by stopping the operation of the cryogenerator.

So that the absorption capacity will not be exhausted too soon, and so that the surface temperature of the absorbing agent - which co-determines the equilibrium pressure in dynamic pump processes - will be as low as possible, the areas of the LT cryosurfaces which are covered with the absorbing agent must be disposed so that they are protected against radiation originating from surfaces at a higher temperature, and so that all gases except He and $H_2$ are previously condensed with higher probability before reaching the absorbing agent.

Even if this precondition is fulfilled, the invention shows - at least in cases where extremely large quantities of gas are not suctioned off, as e.g. in systems for cathode atomization - that as a rule the hydrogen already must be removed at a time when the equilibrium pressure of the other gases at the LT cryosurface has not yet exceeded the permissible values.

As already said, an exception is the use of a cryopump for cathode atomization. Here, such large quantities of gas are generally condensed, that finally the temperature gradient which builds up in the condensed layer, or the plugging of the intermediate spaces between the condensation surfaces force a regeneration. In both cases, the vacuum system up to now had to be stopped for the regeneration. This is a factor that enters into the operating costs, a factor whose influence one would like to reduce as much as possible.

Up to now, regeneration has been accomplished in such a fashion that the cryopump was separated from the vacuum system by a high-vacuum valve and was then switched off. The cryosurfaces then heat up slowly at first, as a consequence of heat irradiation from the environment, and then faster through heat conduction of the gas that is again being vaporized from the condensation surfaces, until room temperature is reached. The liberated gases are pumped off by the fore-vacuum pump, which is also needed to pre-evacuate the vacuum system. Condensed water also vaporizes again, but is partially absorbed at the interior surfaces of the cryopump.

The cooling process can be begun again by restarting the cryogenerator, as soon as a pressure of about 0.1 mbar has again been reached in the cryopump. Here, the partial pressure of water vapor is reduced very quickly to values below $10^{-3}$ mbar. At this residual gas pressure, the heat conduction of the residual gas is only small compared to heat radiation, so that the major portion of the cryogenic power is again available to cool the cryogenerator and the cryosurface.

The time required for regeneration is composed of the heat-up time and of the cool-down time. The heat-up time is determined on the one hand by the amount of gas condensed, and on the other hand by the mass of the HT and LT stage and their associated cryosurfaces. Here, the first variable is determinative with processes that have a high gas pressure, for example in coating systems by means of cathode atomization; the second variable is generally determinative with systems that have a low gas throughput.

The cooling time again depends essentially on the mass of the cryopump that is to be cooled and on the cryogenic power of the two stages in the temperature range that is being traversed. As a rule, a regeneration cycle for an autonomous cryopump takes several hours.

It is the aim of the invention appreciably to shorten the regeneration time of autonomous cryopumps. The inventive method for regenerating the low-temperature condensation surfaces of an autonomous cryopump that is cooled by a cryogenerator is characterized as follows: Before defrosting the gases that have condensed on the condensation surfaces, the thermal contact between the cryogenerator and the condensation surfaces is eliminated.

By installing a device which makes it possible to eliminate and re-establish at will the thermal contact between the LT cryosurfaces and the LT stage of the cryogenerator, the regeneration time in most cases reduced to nearly half, since generally the time for cooling the cryogenerator is the decisive factor, which is obviated by the method according to the invention. Indeed, if it is possible to eliminate the thermal contact between the LT cryosurfaces and the LT stage of the cryogenerator, the LT cryosurfaces can be regenerated without interrupting operation of the cryogenerator, i.e. the LT stage of the cryogenerator continues to remain at a low temperature, and likewise the HT stage with its associated cryosurfaces. Since the revaporization of readily condensible gases such as water vapor and $CO_2$ from the HT surfaces is omitted, the contamination or plugging of the absorption agent by these vapors is avoided. For this reason, it is sufficient to raise the temperature of the LT cryosurface only as much as is necessary to remove the condensed and absorbed permanent gases. However, it is presupposed that the cryogenic power of both stages is adequate to deal with the heat input, which is then mainly determined by the heat conduction of the vaporizing gases.

In a further development of the invention, it is recommended that heating elements be used for the heating process in order to defrost the LT cryosurfaces.

Together with the LT stage, these heating elements are situated in a chamber that is hermetically sealed from the pump chamber. In this way, the vaporization of condensed gases from the cryosurfaces during regeneration no longer depends on heat supplied by radiation and by heat conduction from outside, but is determined practically solely by the heating power of the built-in heating elements. For quick cooling after regeneration, a thermal contact between the LT cryosurfaces and the HT cryosurfaces is furthermore recommended. After the equilibrium temperature has been reached, this contact can again be eliminated. Subsequent cooling to the final temperature takes place by the thermal contact with the LT cryogenerator stage.

With the above-mentioned additional measures, the regeneration time then amounts only to a fraction of the previous time. The associated interruption of operation and production downtime is therefore significantly less.

As regards cryopumps for cathode atomization systems, in which large amounts of gas are condensed, this progress becomes especially clear. Previously, up to 10 hours were frequently required for this, just to remove the condensed amounts of argon. Because it is no longer necessary to cool the large mass of the HT cryosurfaces while restarting the pump, the cooling time is significantly shorter.

The enclosed drawing shows an embodiment of a preferred device to implement thc method. In particular, it shows a cross section through a 2-stage autonomous cryopump in a scale of 1:2.5 with a cryogenic power of about 10 W at 20 K at the LT stage, and about 100 W at 80 K at the HT stage.

In the figure, 1 represents the HT stage and 2 the LT stage of the cryogenerator. The cryosurfaces 3, 4, and 5 make a thermally conducting connecti with the HT stage. The upper cryosurface 3 is formed of concentrically angl copper-plate rings, onto which are soldered 4 radial massive copper webs 7

which emanate from the central copper cup 6. The outer ends of the copper webs are soldered onto the copper cylinder 4 at the point 8. The copper cylinder 4 and the cryosurface 3 thus form a structural unit which is screwed together with the copper base plate 5 at the point 9. The cryosurface 3 is used both to condense the readily condensible gases such as e.g. water vapor, $CO_2$, and higher hydrocarbons, and also to shield the LT stage from radiation incident from the vacuum system. It must have the highest possible conductance for the traversing permanent gases, which are condensed or absorbed at the LT stage.

The LT cryosurfaces consist of plates 10 with V-shaped annular grooves. The latter are connected to the outer cylinder surface of the hollow cylinder 12, by means of a juncture 11. Their interior zones are protected against incident radiation from the HT cryosurface 4 by the ribbing, and are there coated with an absorption agent 13, e.g. activated charcoal.

A heating element is situated within the hollow cylinder 12. It consists of several thin ceramic strips 14 with holes, within which is placed the heating spiral 15. The ceramic strips 14 are distributed along the circumference. Between the heating spiral and the outer tube 16 of the LT stage, there is situated a heat insulator 17 consisting of a heat-proof material. Current is supplied to the heating element through two gas-tight penetrations 47. The current leads 48 are conducted via the baseplate 22 of the cryopump vacuum-tight to the outside.

After the heating element and the insulation have been introduced, the hollow cylinder 12 is welded gas-tight below with an annular part 18. The latter is welded gas-tight, through a spring element 19 of rust-free steel, to a ring 20 which likewise consists of rust-free steel. The ring 20 is soldered onto the baseplate 5.

To reduce heat conduction from the heating element to the LT stage of the cryogenerator, the cavity 12 can be evacuated through the line 21 by means of the cryopump, at the beginning of its cool-down phase, i.e. when the condensation of the permanent gases is not yet taking place. For this purpose, a valve 23 is installed in the baseplate 22. This valve 23 can establish a connection with the cryopump chamber.

The cylindrical copper contact piece 24 is welded or soldered to the upper end of the outer wall of the hollow cylinder 12. The contact piece 24 can be brought into thermal contact at will with the LT stage through 25 or with the HT stage of the cryogenerator through part 6 at the point 26. In the representation shown in the drawing, it is situated in the neutral position. The contact piece 24 is suspended in the crossbeam 27, whose center section is designed as a hollow cylinder 28. The crossbeam is rotatably mounted at one end in a fixed support 29 at the point 30. At the other end, at the point 31, it is rotatably mounted in a movable support 32. This support is brought out through a spring element 33 and thus can be raised or lowered from the outside. Force is transmitted between the crossbeam and the contact piece through the cylindrical intermediate piece 34, which consists of a plastic with low heat conduction, e.g. teflon. The cylindrical ring 28, which forms the centerpiece of the crossbeam 27, transmits the force through the conical ring surfaces 35 and 36, which makes it possible for the contact piece to center itself with respect to the counter surfaces 25 and 26.

The cryogenerator (without the cryosurfaces) can be introduced into the cryopump housing from below, through the cylindrical attachment 37, and can be screwed on vacuum-tight at the point 38. Then the copper plate 5,

which supports the LT cryosurface by means of the spring bellows 19, can be screwed together with the HT stage of the cryogenerator, at the point 39. A seal is here made through a metal gasket 40. Subsequently, the supports 29 and 32 are mounted and the crossbeam 27 is installed and is connected to the supports 29 and 32 at the points 30 and 31. Finally, it is caused to engage the contact piece 24 by means of the Seeger safety 41. Then the shielding 4, which supports the radiation shield 3, is screwed together with the baseplate 5. Finally, the outer jacket 42 of the cryopump is put in place and is screwed vacuum-tight with the baseplate 22. At first the heat insulation 43 may be inserted. This will be discussed later.

The pump stud 44 is used to pre-evacuate the cryopump. At 45 and 46 one can see the connections of the high- and low-pressure lines of the compressor which deliver the helium gas that is necessary to operate the cryogenerator.

The mode of operation of the system will be described in more detail below: Let it be assumed that the cryopump is used for one of the most difficult vacuum processes, namely to generate vacuum in an industrial atomization system equipped with magnetron cathodes. The gas discharge in these systems takes place at an argon pressure of about $3 \cdot 10^{-3}$ mbar. The gas stream that is being pumped off amounts to about 10 mbar liter per second. This corresponds to a condensation enthalpy per unit time of about 4 joules per second. The cryogenic power of the LT stage, namely about 10 W, is then sufficient for stable operation of the LT cryogenerator stage, taking into account additional radiation from the surrounding HT cryosurfaces and the heat conduction of the gases. The like holds for the HT stage, even without the additional heat insulation 43.

If we further assume that argon is preferentially condensed in the outer V-shaped ring zone of the LT cryosurfaces, an argon layer about 0.1 mm thick will grow during one hour of operation. With a spacing of 12 mm between the condensation surfaces, 30 to 40 hours' operation would accordingly be possibly with the given gas flow, without there being any fear that the pumping action of the activated charcoal for hydrogen would be significantly impaired by the condensed argon.

According to the invention, the thermal contact between the low-temperature cryosurfaces and the low-temperature stage is eliminated for regeneration, and the condensed argon and absorbed hydrogen is again vaporized by the built-in electrical heater. After 40 hours' operation, one needs for this about 500 KJ and additionally the energy to heat up the low-temperature cryosurfaces and the heating body to a temperature of at least 130 K (a temperature at which the argon condensed at the absorption agent will also vaporize). Since the weight of these parts amounts to about 1 kilopond, about another 15 KJ are necessary for this. However, it must be assumed that the heat contact between the condensed argon and the support will worsen during the vaporization. For this reason, a larger temperature gradient between the argon and the support will gradually form. This means, the equilibrium temperature of the support will gradually rise, possibly above room temperature. But the energy required for this is still always negligible, compared to the vaporization enthalpy of the argon. Since the heat heat transfer through heat conduction of the gas within the cryopump is independent of pressure, as soon as the free path length is small compared to the wall distances, it is most advantageous if one regenerates at the highest possible gas pressure. The reason for this is not only to shorten the regeneration but also to flush the hydrogen as rapidly as possible with argon. Because its heat conductivity

is 10 times higher compared to argon, a high partial pressure of hydrogen lasting over a longer period of time would cause such high thermal stress on both cryostages that stable operation would no longer be possible, i.e. the temperatures would rise very rapidly and the intended goal of maintaining the basis temperature of the two stages during regeneration could not be achieved. Only when the heat supply to the low-temperature and high-temperature stage is determined only by the heat conduction of argon, in addition to heat radiation, will stable operation be possible.

If nevertheless the time should become too long during which heat conduction of hydrogen dominates and the heat capacity of the high-temperature stage is then no longer sufficient to keep the transient temperature rise low, it is suitable to build in some heat insulation 43 between the walls with a slight spacing, namely between 4 and 42, between which heat transfer will then mainly take place. This heat insulation consists of a double-walled, vacuum-tight welded hollow cylinder of thin sheet metal with poor heat conductivity, i.e. of rust-free steel, whose interior space is evacuated and which suitably is filled up with an absorption agent, for example activated charcoal.

It is not difficult to install a heating body into the cavity 12, with such high heating power that the low-temperature condensation surfaces can be heated in a few minutes. With 1 kW heating power, the vaporization rate theoretically would be e.g. 2500 mbar liter per sec., which can be pumped off by a forepump with 30 $m^3$ per h suction power at a pressure of about 300 mbar. The regeneration would then be completed in 8 minutes. To this one would have to add the cooling time. With such a high heating power, problems of heat transfer between the cryosurfaces and the condensed argon

could arise, and solid argon possibly would be blown out from the intermediate spaces together with the gas stream. Consequently, one limits oneself to lower heating powers of several 100 W. Even then, the regeneration time is shorter by at least one order of magnitude than without these measures.

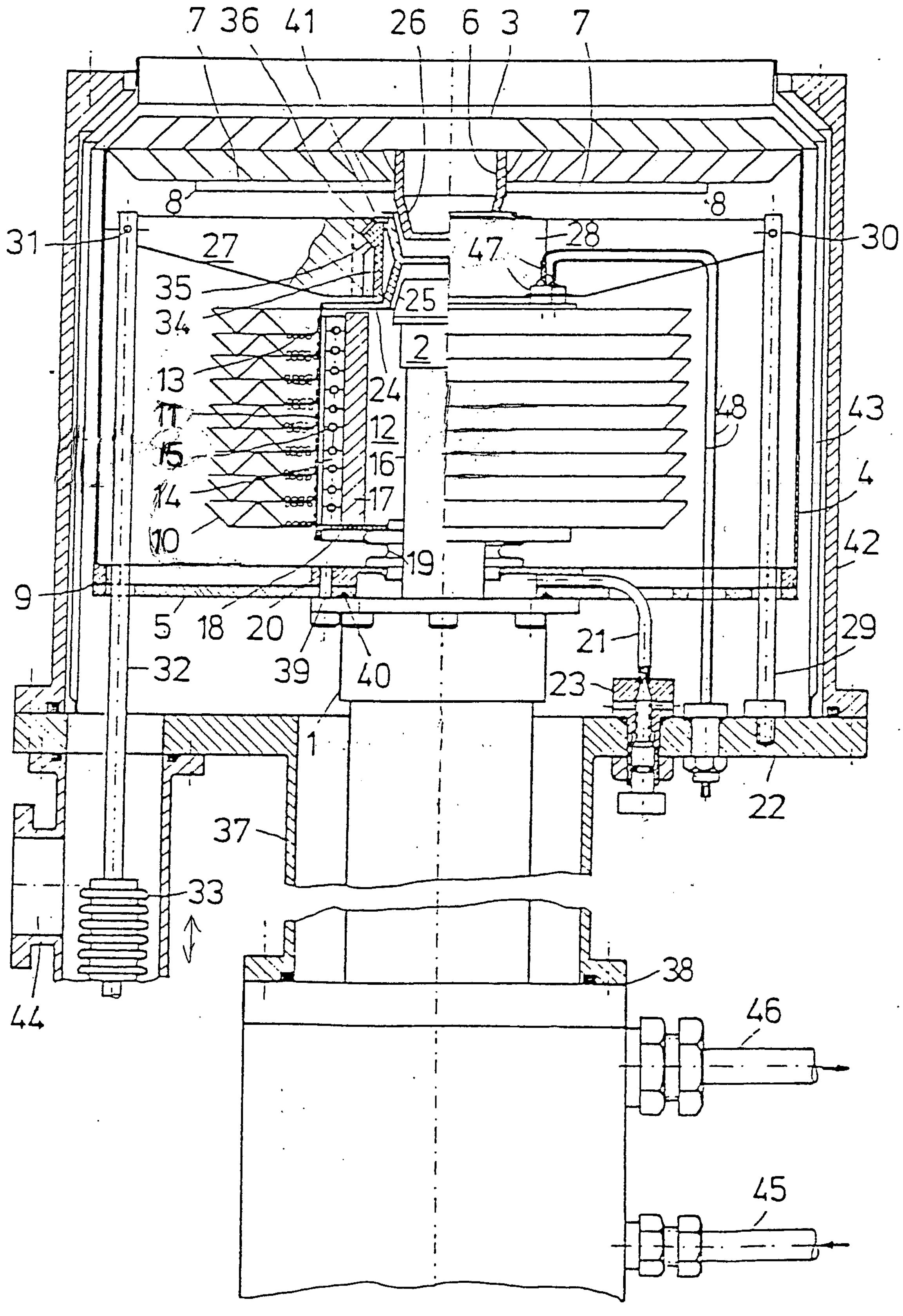
By leaving the cryogenerator in operation during the regeneration process, by eliminating the thermal contact between the low-temperature stage and the low-temperature cryosurface, and by providing the heat for regeneration through a heating device which is characterized that it is not exposed to the possibly explosive atmosphere within the pump, the initial temperatures of both stages of the cryogenerator can practically be maintained. After conclusion of the regeneration, only the mass of the low-temperature cryosurfaces therefore need be cooled.

This can be done in two stages: first by cooling to the temperature of the high-temperature stage through contact with this stage. Because of the large mass of the high-temperature stage, this takes only a few minutes. As soon as this temperature is reached, the residual gas is bound by the absorption agent. The thermal load from heat conduction of the gas vanishes, so that further cooling to the temperature of the low-temperature stage is possible with the lower heating power of about 10 W. For this cooling time also to be as short as possible, it is advantageous that all components of the low-temperature stage be designed so that their heat capacity is a minimum. For example, the cryosurfaces 10 will be fabricated of a thin foil consisting of a high-strength material, such as e.g. hard-rolled rust-free steel sheet, bronze sheet, or hardened nickel or copper alloys. To improve the heat conductivity, these are then provided with a silver coating of $^{1-2}/100$ mm thick-

ness. In this fashion, the cooling time is shortened by a factor of 3 to 4 in comparison to previous regeneration methods. The total time i.e. the sum of the heating time and the cooling time is then less than one hour.

From the above explanations, it is apparent that even when regeneration is essentially limited to the regeneration of the absorption agent, as is the case e.g. with high-vacuum processes, and if the regeneration time is determined mainly by the cooling time, the inventive process entails a considerable time gain. However, in some circumstances it is necessary, if only a forepump is used for the evacuation, in order to lower the hydrogen partial pressure, to flush this with a suitable gas, e.g. with argon, before the cooling process. Sometimes it is sufficient if the forepump is operated with a gas ballast.

The method described above naturally can also be used in conjunction with single-stage cryogenerators, e.g. to operate large absorption pumps. When eliminating the thermal contact between the surfaces that are covered with the absorption agent and the cryogenerator that is still in operation, the required high heating temperatures of several 100°C can be used without needing to fear damage to the temperature-sensitive parts in the cryogenerator.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